

# CAVITATION DAMAGE IN LIQUID METALS

by

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For the Period

20 April - 31 December 1964

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Prepared for

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March 10, 1965

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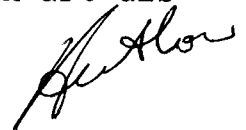
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ABSTRACT

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The operational experience in installing and calibrating a controlled environment test chamber is discussed. This experimental facility has been designed specifically for conducting investigations on the cavitation damage resistance of refractory alloys in pure liquid sodium at temperatures up to 1500°F. An absolute vacuum as low as  $2.5 \times 10^{-3}$  torr and a leak rate as low as  $10 \times 10^{-3}$  torr per hour have been achieved. This facility is capable of maintaining oxygen and moisture levels so as to maintain the test sodium purities less than 50 ppm of oxygen over an eight hour period. Preliminary experiments with 316 stainless steel in 1000°F sodium have shown that the intensity of damage at this temperature is very much lower than that at 400°F. A modified design of the high frequency fatigue specimens has been made and some preliminary experiments have been conducted. A preliminary design for stress corrosion tests has been made. Available data on the mechanical properties of the test metals at high temperatures will be analyzed for correlation with cavitation damage resistance. Plans for the future work are discussed.

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CAVITATION DAMAGE IN LIQUID METALS

Technical Progress Report  
for the Period

20 April - 31 December 1964

I. SUMMARY

This is the first Technical Progress Report prepared under NASA Contract NAS 3-4172, and summarizes the accomplishments of the program for the period.

The object of this program is to qualify candidate materials for use in auxiliary systems for space exploration by the quantitative determination of their resistance to cavitation damage in liquid sodium at temperatures up to 1500°F. Sodium, having two levels of oxygen contamination, 20 and 200 ppm, will be used in the experiments. The magnetostriction apparatus, operating in a controlled environment chamber, will be employed in the cavitation damage tests. These tests will be augmented by corrosion fatigue and stress corrosion cracking tests, which will be performed on two of the alloys of primary interest: 316 stainless steel and TZM alloy.

The major part of the program to date has been concerned with obtaining reproducible and reliable operation of the test facility at specified sodium purity levels. Effort has also been devoted to the design of the corrosion fatigue and stress corrosion cracking specimens to be used in this program. Cavitation damage experiments have been started for 316 stainless steel at 1000°F. These experiments have shown that the damage intensity at 1000°F is very much lower than that at 400°F.

## II. INTRODUCTION

Very little is known about the prediction of damage caused by a cavitating flow regime such as found in high speed turbomachinery. With the advent of auxiliary power generating systems for space vehicles, these problems become very important. Due to the minimum weight designs of these fluid circuits, high velocity flow will be incurred which in turn may lead to much greater cavitation damage intensity than that normally observed on an earthbound counterpart. In addition, high efficiency systems are believed attainable if liquid metals are used as both the thermodynamic and heat transfer fluids. The use of liquid metals adds many more parameters to the investigation of cavitation damage because of their corrosion and mass transfer tendencies.

Cavitation damage investigations using liquids such as water have taken advantage of many types of equipment from laboratory devices such as a rotating disc to actual field turbomachinery. Due to the many precautions which must be taken with liquid sodium, experimental investigations using actual turbomachinery are extremely tedious. Hence, the laboratory devices such as the cavitating venturi, rotating disc and magnetostriction oscillator offer the most practical solutions to the question of how to test the cavitation damage resistance of materials. The magnetostriction oscillator satisfies the following criteria:

(1) Produces sufficient intensity to permit relatively short testing times.

(2) Operates simply, precisely, and reliably to yield frequent and reliable data with little shut-down.

(3) Compact size allows operation in a minimum volume of liquid metal thus making all phases of liquid metal handling and cleaning easier.

On the basis of the above criteria, the magnetostriction apparatus was chosen as the testing apparatus to be used in this program.

The apparatus used by HYDRONAUTICS, Incorporated under an earlier NASA contract (NASr-105) has been described in detail in earlier reports (1,2). On the basis of these early tests, it was concluded that a more detailed analysis could be accomplished on a more sophisticated level if new equipment could be used. Consequently, a new laboratory apparatus was designed and fabricated.

The objectives of this program are two-fold. The first is the determination of the cavitation damage behavior of selected refractory metal alloys and 316 stainless steel in liquid sodium at temperatures up to 1500°F. The second objective is to investigate methods for predicting cavitation damage in actual operating equipment by correlating accelerated laboratory tests with data available under real time conditions.

In a study such as this many variables exist, such as:

- (1) The effect of liquid metal physical properties over the temperature range and the purity of the liquid metal.
- (2) The effect of the test material properties over the temperature range.
- (3) The effect of the atmosphere over the liquid metal.

Each of these variables could be the single subject of interest in a research program; hence one of the purposes of this current program is to define these variables.

Therefore, these experiments are primarily aimed towards qualifying candidate materials for use in the aforementioned auxiliary power systems for space exploration. The five alloys of interest are listed in Table 1. The properties and characteristics of each of these alloys will be covered in more detail later in the report, but as a general statement it may be said that all of these alloys exhibit to varying degrees, high strength and other desirable mechanical properties at elevated temperatures. Each alloy selected has a different base metal as its prime constituent. The selection of materials in this manner allows for the screening of typical alloys of a refractory group (except for the 316 stainless steel which is used for comparison purposes) so that promising materials can be identified rapidly for more detailed studies in future programs.

To gain further insight into the properties of a material which might identify it as being resistant to cavitation damage, high frequency fatigue and stress corrosion cracking tests will

be made on two of the alloys of interest, TZM and 316 stainless steel. Both of these tests will be conducted in a liquid sodium environment such as that used for the cavitation damage tests.

### III. CALIBRATION OF THE TEST FACILITY

The test facility, Figure 1, for conducting cavitation damage experiments on refractory metals in high temperature alkali liquid metals was designed and fabricated under the previous Contract NASr-105. The details of design and the general specification requirements were reported earlier in References 1 and 2.

During the present investigation, the test facility with all its interconnecting piping, electrical wiring and instrumentation was assembled, tested, and calibrated. The final qualification of the facility required that the purity of sodium used in the experiment, be maintained at less than 20 ppm of oxide over an eight hour period under normal testing conditions. To attain this objective of sodium purity, considerable time was spent in checking-out the minute details of operation and performance. Leaks in the vacuum and cover gas systems were particularly elusive to find and remedy.

It is believed that this facility is unique in that it is the first magnetostriction device, with all the auxiliary systems self-contained, specifically designed to perform cavitation damage tests in liquid sodium at elevated temperatures. Despite the many design features of the facility considerable time is required to obtain controlled experimental conditions. The sequence

of steps involved in a normal experimental run over an eight hour period is given in Table 2. Many other operations in addition to those listed in Table 2 must be done, such as logging operational and other experimental data, cleaning and weighing of specimens, and maintaining a continuous watch on all instruments which are monitoring the experiment. As experience and confidence with the facility increase, such tasks as taking the leak rate daily may be circumvented, if pump down pressures appear normal. Also, the addition of a timing switch which will allow the hot trap to cool before the normal working day begins will also serve to increase the actual testing time attainable in the argon chamber.

It is worth reviewing the major difficulties encountered with the various systems of the test facility and the means taken to correct faulty operation or performance.

#### A. Vacuum System

The vacuum chamber was designed to be evacuated to an absolute pressure of  $2.5 \times 10^{-3}$  torr of mercury in a reasonable period of time (one to two hours when the chamber is dry and clean). A pressure increase not to exceed  $15 \times 10^{-3}$  torr/hour was specified. These figures were achieved for the stripped down vacuum chamber as delivered by the fabricator. The addition of the auxiliary piping systems for cover gas and sodium, which were required for the operation of the integrated facility, increased the incidence of leaks beyond that which could be tolerated. Therefore, systematic leak detection and correction had

to be undertaken. The specified pressures were achieved, after an exhaustive search for minute leaks at the numerous seals and valves. High temperature gaskets and dynamic O-ring seals proved to be particularly troublesome.

In many areas, the design sophistication of the facility was confined by the state-of-the-art. General examples are listed below.

1. O-ring seals for the retort flange would reach 1200<sup>0</sup>F when the retort was heated to 1500<sup>0</sup>F. This problem was solved by using silver plated Inconel O-rings and by using 316 stainless steel bolts to reduce differential permanent set due to thermal cycling.

2. Addition of band heaters to the specimen lock caused local over-heating of buna O-rings and gave rise to leaks in the specimen lock and in the box. After several trials the capacity of the heaters was reduced until the O-rings performed satisfactorily.

3. The cover gas system as designed originally gave rise to too many leaks in the fittings. This problem was solved by eliminating several fittings, thereby decreasing the possibilities for leaks which, of course, eliminated several safety features such as regulators and driers. Present experience indicates that with care these safety features are not necessary.

4. The Pirani (thermocouple type) vacuum gauges used in the facility were found to be inadequate for the purpose in spite of repeated calibration and factory adjustment. This

problem was solved by using a McLeod gauge for determining absolute vacuum. The Pirani gauges are still used to determine leak rates in the dry box.

5. Leak detection in the various components was an elusive process requiring considerable time. A helium leak detector, operated by a trained technician, was used to locate the final leaks.

#### B. Cover Gas System

The next important system which required considerable design modification, adjustment and check-out was the cover gas system. A static cover gas system was specified for simplicity and reliability inasmuch as commercial sources for high purity argon were available. Some problems associated with the cover gas system are listed below.

1. The O<sub>2</sub> analyzer and the hygrometer gave several initial troubles because of the specialized nature of their use for this particular case. These analyzers are mainly intended for use with positive pressures at both inlet and outlet. Since the dry box cannot be pressurized, sampling of the argon at various time intervals required a vacuum pump to pull the argon from the test chamber, and through the analyzers. After several trials, satisfactory sampling circuits for both the O<sub>2</sub> meter and the hygrometer were perfected. During these trials, the components of the meters had to be replaced several times and the meters had to be recalibrated.



2. The cover gas piping system was modified to reduce the possibility of leaks as pointed out earlier. The original cover gas piping circuit was shown in References 1 and 2.

3. When the glove port covers are removed, exposing the PVC gloves to both room and dry box atmospheres, diffusion of moisture and oxygen through the gloves is a major problem imposed by the state-of-the-art. There are butyl gloves available with much lower moisture and oxygen diffusion rates. However, these gloves cannot be used because the sulphur introduced into the butyl during vulcanization is released into the dry box when the box is under vacuum. Apart from the inherent difficulty of high diffusion rates through the PVC gloves (3), the quality of manufacture is also not very encouraging. Several gloves had to be returned due to manufacturing defects. Figure 3 shows that for reasonable glove exposure times the  $O_2$  and  $H_2O$  levels in the box can be kept to acceptable values, less than 50 ppm total.

#### C. Sodium Transfer System

The unique feature of this magnetostriction test facility is the self-contained nature of sodium storage, purification (hot trapping), and transfer. A detailed description of this system can be found in References 1 and 2. Reactor grade sodium is purchased in 200 pound stainless steel storage drums having maximum oxide contamination of 50 ppm. Twenty-five pound batches of this sodium are transferred into a specially designed hot

trap containing zirconium chips. The hot trap is heated continuously at  $1400^{\circ}\text{F}$  to remove oxides in the sodium to below 10 ppm. After hot trapping, the sodium is transferred to the test retort where the cavitation experiments are performed. One drum of sodium will fill the hot trap approximately 8 times. Each hot trap of sodium yields approximately 13 retort fillings.

Problems associated with this system are:

1. Transfer line blockage due to residual oxide and carbon deposits and/or due to non-uniform heating of the transfer lines.
2. Sodium vapor blockage of the vacuum and gas lines entering the hot trap due to condensing of sodium vapor in these lines. This requires periodic cleaning. No reliable vapor trap is yet available.

#### D. Heating and Cooling Systems

The retort system incorporated into the bottom of the test facility is operated at temperatures up to  $1500^{\circ}\text{F}$ . At the same time the working surfaces of the box have to be maintained below  $150^{\circ}\text{F}$ .

Much of the cooling water valving supplied by the dry box manufacturer was eliminated in order to obtain the desired flow rates through the false bottom of the dry box. It was also found that improper fitting of remote temperature indicating bulbs in the line resulted in blockage of flow; this too was corrected.

These modifications resulted in much more acceptable temperature distributions across the bottom of the dry box, as shown in Figure 4. A sheet metal glove shield around the retort nozzle is used for high temperature experiments to further protect the operator. Difficulty is still experienced with the O-ring in the retort flange nozzle used to seal the elevating head (a sliding fit). At the higher temperatures, the O-ring becomes hard and brittle and the male fitting on the elevating head occasionally breaks the O-ring as it slides through, thereby destroying the vapor and vacuum seal and contaminating the sodium with carbon debris. This problem could be eliminated by using a metal O-ring, which currently is a non-standard commercial item. Presently the O-ring is replaced periodically when it begins to show wear.

The electrical contacts of the retort furnace have been found to oxidize under high temperature, thereby reducing the ultimate temperature due to the increased contact resistance formed across the low voltage connections. Periodically these contacts must be cleaned in order to maintain efficient service. In addition, more insulation had to be added to the exterior of the pot-type laboratory retort furnace to prolong its useful life.

#### E. Sodium Analyses

The final calibration of the dry box required that contamination of sodium samples in the retort be maintained below 20 ppm over an 8 hour experimental run under normal operating

conditions. Commercial analysis for oxygen level in sodium by the relatively standard method of mercury amalgamation was considered, but due to the high cost, inherent experimental difficulties and long delays in reporting results, this method of analysis was deemed unsuitable for use on this program. NASA specified that the recently developed neutron activation technique, which was available as a commercial service, be used for oxygen analysis of the sodium.

Briefly, the technique works on the following principle: Properly prepared samples of sodium are exposed to high energy fast neutrons for a fraction of a minute, where upon the formation of the isotope,  $N^{16}$ , is induced by the  $D^{16} (n,p) N^{16}$  reaction. The amount of  $N^{16}$  formed is directly proportional to the oxygen in the sample. Immediately after irradiation, the sample's radioactivity is measured and analyzed by multi-channel gamma ray spectrometry.

There are some important advantages of the neutron activation technique over the mercury amalgamation technique. Neutron activation can be done quickly and is relatively inexpensive; further, the sample remains sealed in its container throughout the analysis. The lower limits of sensitivity are reported to be 30 micrograms of oxygen per sample with a single irradiation precision of about  $\pm 30$  percent of the value (4). For a five gram sample, this would be 6 ppm. However, the irradiating target strength, counting rates, operator error and the contamination within the container, can cause large deviations from the mean reading. Other investigators (5) have reported that oxygen

detection below 10 mg is difficult and only with relatively large samples of 100 gms can analyses be made to 10 ppm  $\pm 15$  percent. This will be discussed more fully later in light of analyses made.

The sampling technique was difficult and had to be perfected after several trials. The difficulties encountered in sampling sodium were solved as follows:

1. Initially a 1/8" O.D. 316 stainless steel tube was fitted into the retort head through a compression seal fitting. The sodium was pumped from the retort through this pipe by pressurizing the retort. This method proved to be tedious because of the necessity to heat the sampling tube so that the sodium would not solidify in the tube. Since it was difficult to control the temperature of the tube and the pressure in the retort simultaneously, the sodium occasionally overflowed the sample capsule into the dry box. This method was discarded in favor of a simple dip tube which allows more precise capsule filling but may introduce greater contamination to the sample. However, several dip tubes are used, each one cleaned thoroughly before and after each sampling.

2. The sample capsule is welded in the dry box using a tungsten electrode. This welding technique required some development time. Any sodium wetting of the closure to be welded would cause the welding operation to be ineffective, therefore, filling the capsule required extreme care. Also the heat generated during welding would expand the sodium sample in the capsule causing it to overflow before the weld seal was completed. This problem was overcome by increasing the size of the heat sink to a six inch cube of copper.

To date, the results obtained by the neutron activation analyses leave much to be desired. Table 3 lists the data obtained thus far. In the case of samples 1, 2, 3, 5, 6, 6B and 7, the oxide content apparently exceeds the saturation limits for sodium oxide in sodium (Figure 5) and are at least an order of magnitude above a random sample analysis from the same retort of sodium. This random sample check by NASA yielded 80 ppm of oxide contamination. The results of the eight-hour sampling varied from 269 to 507 ppm of  $O_2$  in a random fashion despite the continuous increase in cover gas oxygen and moisture contamination from 12 to 40 ppm and 46 to 100 ppm, respectively. Independent NASA tests on samples taken at the beginning and at the end of the run yielded  $O_2$  values of 34 and 35 ppm, respectively. Samples 43-51 show a general downward trend in  $O_2$  contamination but the actual values are still suspect.

#### F. Magnetostriction Assembly

A detailed study of the characteristics of the magnetostriction transducer system was made, to determine the frequency at which the best quality factor ( $Q$ , defined as the ratio of usable energy stored to the total input energy) is obtained for cavitation damage and high frequency fatigue studies in liquid sodium. This is especially important for the fatigue experiments since the transducer must be operated at or near the optimum  $Q$  because of the increased damping of metals at higher temperatures.

The procedure undertaken was as follows: The resonant frequency of the transducer-velocity transformer combination can be varied by changing the length of the extension rods attached to

the free end of the velocity transformer. The relationship between the amplitude (pick-up coil voltage) and the frequency is experimentally determined for various resonant frequencies as shown in Figure 6 for approximately constant power input. From these data the quality factor for each resonant frequency was determined by the following formula:

$$Q = \frac{1}{\alpha} = \frac{\pi}{\sqrt{3}} \frac{\Delta f}{f_n}$$

where

- Q is the quality factor,
- $\alpha$  is the logarithmic decrement,
- $\Delta f$  is the band width at half the maximum amplitude, and
- $f_n$  is the resonant frequency.

The value of Q for various resonant frequencies has been plotted as a function of  $f_n$  in Figure 7. The design of the high frequency fatigue specimens was also based on an operating frequency of 14.0 kcs to obtain a near optimum quality factor.

The operation of the magnetostriction oscillator at temperatures up to 1500°F (using a 316 stainless steel velocity transformer) has been proven to be feasible. In this connection, minor difficulty was experienced early in the program with loss of the oscilloscope display voltage which monitored the amplitude of the transducer. This trouble was diagnosed as loss of magnetic strength in the pick-up coil, permanent magnet system. The above condition was corrected by installing a more powerful magnet with a higher curie point.

#### IV. TEST PROGRAM

##### A. Cavitation Damage Testing

The purpose of this phase of the program is to determine quantitatively the cavitation damage resistance of the five (5) candidate alloys of interest. The primary interest will be in refractory alloys, using 316 stainless steel for comparison purposes. The operating temperatures of interest are 400°F, 1000°F, and 1500°F. The effect of temperature on cavitation damage resistance of 316 stainless steel, over a range of temperatures from 300°F to 1500°F, will also be studied. The effect of oxygen impurities in the sodium will be evaluated at two levels of impurities, less than 20 ppm and  $200 \pm 50$  ppm.

All the metals with the exception of Cb-132M have been machined for cavitation damage specimens. Experiments using 316 stainless steel have begun at a sodium temperature of 1000°F. Two specimens have been tested; one specimen for thirteen hours and the other for 25 hours. Both of these buttons exhibit damage patterns, but as yet no weight loss is detectable. Figure 8 shows the progression of damage with test time on the surface of one specimen. These tests have shown that the intensity of cavitation damage is very much lower than that observed in 400°F sodium. Further experiments are in progress to determine the intensity of damage as a function of temperature using 316 stainless steel.



B. High Frequency Fatigue Testing

High frequency fatigue testing in a magnetostriction oscillator is being performed on 316 stainless steel and TZM along with stress corrosion cracking studies, to augment the cavitation damage testing. Materials undergoing cavitation damage are subjected to high strain rates. These rates can be duplicated in high frequency fatigue data. In addition, values for the yield and ultimate tensile strengths can be obtained from high strain rate fatigue data and these values can be translated into strain energy data, thereby making a correlation between strain energy and cavitation damage rate more meaningful.

Figure 9 illustrates a typical high frequency fatigue specimen to be used in this program. Other types of high frequency fatigue specimens have been proposed earlier (1). In one design, the taper needed to produce a sufficient stress to fracture the metal proved either too costly or too difficult to machine for the alloys of interest. Hence, design attention was focused on a notch-type specimen due to the ease and low cost of machining reproducible samples. The design of a notch-type specimen is as follows:

1. The exponential horn assembly is made to oscillate at 14.0 kcs by adding a 1 3/4" straight 316 stainless steel rod to the assembly.

2. Then a 3/8" diameter specimen rod of the material of interest is attached to the transducer and lowered into the sodium.

3. Various lengths of this rod are vibrated until the proper length is found to bring the natural frequency of the system to 14.0 kcs.

4. This is the half wave length at that frequency for the desired temperature.

From the preceding steps, the velocity of sound,  $c$ , may be determined by:

$$c = \lambda \cdot f_n$$

where  $\lambda$  is the wavelength and  $f_n$  is the resonant frequency.

From this value of  $c$ , the modulus of elasticity also can be determined for future stress calculations by:

$$c = \sqrt{\frac{E}{\rho}}$$

where  $E$  is the modulus of elasticity and  $\rho$  is the material density.

After the half wavelength of vibration of the material at the frequency and temperature of operation is determined, a notch must be made at the vibrational node so as to increase the stress at this point. The notch geometry must be selected so as to keep the notch sensitivity at a minimum. The notch sensitivity is defined as:

$$\eta = \frac{k_f - 1}{k_t - 1}$$

where

$$k_f = \frac{\text{unnotched fatigue strength}}{\text{notched fatigue strength}}, \text{ and}$$

$$k_t = \text{theoretical stress concentration factor.}$$

The strains produced in the actual specimen at the notch can be calculated by:

$$\epsilon_{\max} = \frac{2\pi f_n}{c} \xi_{\max}$$

where

$$\epsilon_{\max} = \text{strain at the notch,}$$

$$f_n = \text{resonant frequency, and}$$

$$\xi_{\max} = \text{vibrational amplitude.}$$

High frequency fatigue specimens of 316 stainless steel for testing in sodium at 1000°F are currently being machined and will be tested shortly. In a preliminary test performed in order to verify the design, a specimen began to fail after 1 1/2 minutes at a stress level of 55,000 psi and frequency of 14.0 kcs. Complete failure occurred after four minutes. This specimen is shown in Figure 10.

C. Stress Corrosion Cracking Testing

Preliminary studies on stress corrosion cracking will be made on two metals. The purpose of these preliminary studies is to determine whether the susceptibility of a metal to stress corrosion cracking can be used as an index for its cavitation damage resistance. The metals of interest are 316 stainless steel and TZM.

There are two methods in normal use for producing stress corrosion cracking: constant strain and constant load. Under constant strain the specimen is loaded to a fixed value and upon cracking the stress is relieved, thereby tending to reduce the rate of cracking. In the constant load device, as cracking develops the stress is increased due to a decrease in cross-sectional area at the crack. At this point, the damage rate is increased and therefore is readily identified.

The first method (constant strain) employs a typical horseshoe shaped specimen or bent beam in a fixed holder, while the second method generally employs a tensile type specimen loaded by weights. The susceptibility of a metal to stress corrosion cracking can be readily demonstrated by the first method, while a parametric study of the amount of stress versus time to fail is more easily obtained with a constant load apparatus.

Figure 11 shows a proposed stress corrosion specimen which is a modification of the horseshoe type in which constant strain is applied by closing the gap of a split ring. The maximum tensile stress occurs on the external surface along an axis line

opposite the gap opening. From classical elastic theory, the stress and deflection can be calculated with a fair degree of certainty. However, under high temperature exposure, allowance must be made for expansion of the specimen and holding bolt (same material as the specimen) as well as loss of elastic properties, in determining actual stress levels. Several sample specimens will be made to determine the feasibility of translating a known stress level in the ambient temperature condition to a preselected stress level at the high temperature condition. If this proves feasible, several specimens will be exposed statically to liquid sodium at 1000°F and 1500°F at several stress levels and examined periodically (every 60 hours) for cracks and other signs of degradation.

## V. ANALYSES OF DATA

### A. Metals to be Tested

With the exception of 316 stainless steel, the metals of interest in this program are those of the newly developed refractory group. Table 1 lists the chemical composition of these candidate materials. Due to their relatively recent development a brief review of each is useful:

(1) 316 Stainless Steel - An 18 percent Cr, 8 percent Ni, Mo type stainless steel, particularly suited for high temperature service. It is similar to type 304, except that the addition of molybdenum greatly increases its creep strength at elevated temperatures. It is useful in high strength service up to approximately 1500°F and resistant to oxidation up to 1650°F.

(2) Stellite 6B - A cobalt based alloy with a relatively low (1.1 percent) carbon content, making it the easiest of the cobalt based alloys to form and work.

(3) TZM (Titanium-Zirconium-Molybdenum) - A 0.5 percent Ti, 0.1 percent Zr, Mo based alloy, similar to the more common Mo-0.5 Ti, but having greater hot strength and higher recrystallization temperature. Elevated temperature strength is brought about by a combination of strain hardening and precipitation of complex carbides causing dispersion hardening.

(4) T-222 (Ta-10W-2.5 Hf) - Similar to T-111 (Ta-8W-2Hf) but it exhibits superior strength from approximately 2000°F to 3500°F. Retains ductibility for easy forming. Excellent corrosion resistance probably due to hafnium barbitides.

(5) Cb-132 M - This alloy is columbium based, modified with reactive metals such as hafnium or zirconium to minimize alkali metal corrosion attack. This particular alloy is to be supplied, along with information on its properties, by NASA.

As in any newly developed alloy, the data available to users of the refractory metal alloys are limited. These alloys are marked by their body-centered cubic structure and transition from brittle to ductile behavior over a relatively narrow temperature range. The columbium and tantalum alloys derive their strength by forming solid solutions at the higher temperatures, whereas the molybdenum based alloy (TZM) derives its strength from strain hardening and complex interstitial carbides at higher

temperatures. All of the refractory metals oxidize catastrophically in air atmospheres at elevated temperatures, in some cases leading to embrittlement of the base metal or formation of volatile oxides which burn in air (Reference 6). For this reason, the effect of different oxygen purity levels of sodium shall be studied to see if oxygen contamination of sodium affects the cavitation damage resistance of these metals.

As stated in earlier reports (References 1 and 2) it is believed that a correlation exists between the strain energy of a material and its resistance to cavitation damage. Complete stress-strain curves to fracture at temperatures of interest of even common metals are difficult to obtain. This problem becomes still more acute when considering refractory alloys, hence the strain energy,  $S_e$ , for a given temperature, is estimated by the following formula:

$$S_e = \frac{(Y.S. + T.S.)}{2} \epsilon$$

where

Y.S. is the yield strength,  
T.S. is the ultimate tensile strength, and  
 $\epsilon$  is the strain.

Figures 12, 13, 14 and 15 show the three mechanical properties (yield strength, tensile strength, and elongation) of four of the five alloys of interest as functions of temperature. Based on these data and the general formula presented for strain energy, Figure 16 was prepared. This shows the estimated strain energies of each of the four alloys as functions of temperature. It is expected that the experimental data obtained on the cavitation damage resistance of these metals may be related to their strain energies.

## VI. FUTURE WORK

### A. Sodium Analyses

Statistically representative samples will be sent to NASA-Lewis Research Center for analysis by a modified mercury amalgamation technique.

### B. Cavitation Damage Tests

Experimental investigations on the cavitation damage resistance of 316 stainless steel in sodium at various temperatures from 300 to 1500°F at 100° intervals will be continued. It is planned to test the cavitation damage resistance of all the materials listed in Table 1 at sodium temperatures of 400°F, 1000°F and at 1500°F.

### C. Corrosion Fatigue Tests

Final designs of the 1500°F 316 stainless steel fatigue specimen and the 1000°F and 1500°F TZM fatigue specimens will be made and appropriate fatigue specimens will be fabricated. The



1000°F 316 stainless steel fatigue specimens are ready and will be tested as soon as cavitation damage data are collected.

D. Stress Corrosion Cracking Tests

Several 316 stainless steel and TZM ring specimens will be prestressed and tested statically in liquid sodium at 1000°F and 1500°F, over week-end periods when the cavitation apparatus is not used. This will be done as soon as the analysis of the induced stress levels at high temperature can be made.

E. Analyses of Data

A systematic analysis will be performed on the data obtained from the various tests which will be run during the quarter. It will be determined whether the mechanical properties of the various alloys tested in this period can be related to the cavitation damage resistance of the alloys. The data obtained from corrosion fatigue and stress corrosion cracking studies will, along with the physical properties of the liquid metal and its corrosion and mass transfer tendencies, be used to augment the analyses.

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1. H. S. Preiser, A. Thiruvengadam and C. Couchman, III, "Cavitation Damage in Liquid Sodium," HYDRONAUTICS, Inc., NASA CR-54072, April 1964.
2. H. S. Preiser, A. Thiruvengadam, and C. Couchman, III, "Cavitation Damage Research Facilities for High Temperature Liquid Alkali Metal Studies," ASME Symposium on Cavitation Research Facilities and Techniques, Philadelphia, Pa., May 1964.
3. J. E. Ayer, D. R. Schmidt, and R. M. Mayfield, "Permeation of Water Vapor Through Polymeric Films," Journal of Applied Polymer Science, 3, 7, 1-10 (1960).
4. "Neutron Activation Analysis Procedures for Determination of Oxygen in Potassium," General Dynamics Corp., NASA CR-54100, January 1965.
5. L. Steele and W. Meinke, "Determination of Oxygen by Activation Analysis with Fast Neutrons Using a Low-Cost Portable Neutron Generator," Analytical Chemistry, 34, 185, (1962).
6. C. B. Jackson, ed., "Liquid Metals Handbook, Sodium (Na) Supplement," Third Edition, Supt. of Documents, Washington, D.C., TID 5277, July 1955.
7. "Handling and Uses of the Alkali Metals," American Chemical Society, Washington, D. C., Page 64, 1957.

TABLE 1  
Nominal Chemical Compositions of Test Metals  
Weight Percent

Material	C	Mn	P	S	Si	Cr	Ni	Mo	Fe
316 S.S.	.08	2.0	.03	.03	.75	16-18	11-14	2-3	Balance
	C	Ti	Zr	O <sub>2</sub>	H <sub>2</sub>	N <sub>2</sub>	Ni	Mo	Fe
TZM	.016	.48	.08	<.0002	<.0001	.0003	<.001	Bal.	<.001
	W	Hf	C	Ta	O <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>		
T-222	9.02	2.72	.007	Bal.	.0019	.0037	.00012		
	Ni	Si	Fe	Mn	Cr	Mo	W	C	Cobalt
Stellite 6B	3	2	3	2	30	1.5	4.5	1.1	Bal.
	Ta	W	Mo	Zr	C	Cb			
Cb-132 M	20	15	5	1	.1	Bal.			

TABLE 2

Facility Operation Over An Eight Hour Period

<u>Time</u>	<u>Task</u>
0830	Dry box has been under vacuum over night while sodium has been hot trapped at 1400°F. Secure vacuum valves to air lock, specimen lock and main chamber. Heat transfer lines. Start up O <sub>2</sub> and H <sub>2</sub> O analyzers for sampling cover gas manifold. Reduce hot trap temperature for transfer. Evacuate retort.
0915	Determine leak rate into main chamber. If less than 15 μ/hr, then back flush box with cover gas.
0935	Bring cover gas to atmospheric pressure. Switch analyzers to monitor box atmosphere.
1000	If analyzers are reading less than a total of 5 ppm of O <sub>2</sub> and H <sub>2</sub> O, transfer sodium to retort and heat to test temperature.
1100	Begin four hours of cavitation testing on selected specimens. Weighings are taken hourly.
1500	Complete last cavitation test. Sample sodium at test temperature. Cool sodium for transfer to dump drum.
1600	Dump sodium and allow sodium vapor in box atmosphere to condense.
1630	If all sodium vapor in box atmosphere has condensed, pull vacuum over night and secure all other systems.
1700	Maintain hot trap temperature at 1400°F, over night.

TABLE 3

Oxygen Analyses of Sodium Samples  
by the Neutron Activation Technique

No.	Sample Type	Cover O <sub>2</sub>	Gas (ppm) H <sub>2</sub> O	Na Temp. °F	Oxygen ppm	Remarks
00	Blank	~	~	~	26.7	For purpose of testing weld closures
01	Blank	3	30	~	26.4	
02	Blank	~	~	~	30.7	
05	Blank	3	60	~	30.2	
06	Blank	8	85	~	40.6	
1	Sodium	3	30	950	3220*	Siphon Transfer, cap welded.
2	Sodium	~	~	1000	6800*	Siphon Transfer, cap welded.
3	Sodium	~	~	1000	5570*	Siphon Transfer, cap welded.
5	Sodium	3	60	1000	7450*	Siphon Transfer, cap welded.
6	Sodium	8	85	1000	6300*	Siphon Transfer, cap welded.
6B	Sodium	14	45	450	3140*	Na transferred at 400°F and sampled at 450, 600, and 1000°F by Siphon transfer. Cap welded. Note: NASA analyzed a random sample by mercury amalgamation methods and reported a oxygen content of 80 ppm.
7	Sodium	14	90	600	3140*	
8	Sodium	14	120	1000	1810*	

\* Based on average tare weight of capsule of 28.6 gm.

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TABLE 3 (Continued)

No.	Sample Type	Cover O <sub>2</sub>	Gas (ppm) H <sub>2</sub> O	Na Temp °F	Oxygen ppm	Remarks
1A	Blank	~	~	~	25.0 ± 2.2	
3A	Na (4.3561g)	14	45	400	98.7 ± 18.4	Dip tube transfer, cap welded.
4A	Na (1.8393g)	19	58	400	3σ < 81.5	Dip tube transfer, cap welded. Na weight too small for O <sub>2</sub> detection, 3σ of 81.5 applied.
5A	Blank	28	55	~	30.2 ± 2.5	Dip tube transfer, welded crimp.
6A	Na (2.8227g)	~	~	400	67.3 ± 24.8	Dip tube transfer, welded crimp.
7A	Na (2.2143g)	~	~	400	221 ± 45	Dip tube transfer, welded crimp.
9	Na (3.3042g)	14	45	1000	115 ± 30	Dip tube transfer.
10	Blank	19	58	~	27.5 ± 3.5	
11	Na (3.3304g)	28	56	1000	39.0 ± 27.0	Dip tube transfer, Na 1000°F.
12	Blank	24	34	~	29.1 ± 3.0	
13	Na (3.3848g)	23.5	42	1000	148 ± 30	Dip tube transfer, Na 1000°F.
14	Blank	24	46	~	27.1 ± 3.0	

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TABLE 3 (Concluded)

No.	Sample Type	Cover Gas (ppm) O <sub>2</sub> H <sub>2</sub> O	Na Temp. °F	Oxygen ppm	Remarks	
28	Blank	12	46	~	25.5 ± 1.7	Sample numbers 28 through 41 are the results of an eight (8) hour sampling run to determine the effect of time on the purity of sodium in the dry box retort. Note: additional samples at the beginning and end of the run were sent to NASA for analysis by mercury amalgamation methods. The oxide contamination in the capsules was 34 and 35 ppm, respectively.
29	Na (3.5913g)	12	46	1000	278 ± 22	
31	Na (4.2814g)	15	69	1000	269 ± 21	
32	Na (5.2474g)	24	78	1000	507 ± 23	
34	Na (5.1991g)	25	80	1000	487 ± 23	
35	Na (4.1662g)	30	78	1000	319 ± 24	
36	Na (4.8946g)	35	84	1000	490 ± 25	
37	Na (4.1983g)	38	90	1000	381 ± 26	
39	Na (4.7055g)	42	97	1000	340 ± 23	
40	Na (4.7504g)	40	100	1000	444 ± 25	
41	Blank	39	100	~	22.1 ± 2.1	
43	Na (4.6018g)	12	6	1000	77.8 ± 28.1	Spec. #1, 316 SS Start of day End of day
44	Na (4.8235g)	50	30	1000	89.2 ± 29.0	
46	Na (4.7370g)	20	3	1000	83.4 ± 28.5	Spec. #2, 316 SS Start of day End of day
47	Na (4.6211g)	78	72	1000	138 ± 29	
48	Na (5.3541g)	7.6	2.6	1000	60.7 ± 24.6	Spec. #2, 316 SS Start of day End of day
51	Na (4.8187g)	50	63	1000	73.9 ± 26.7	

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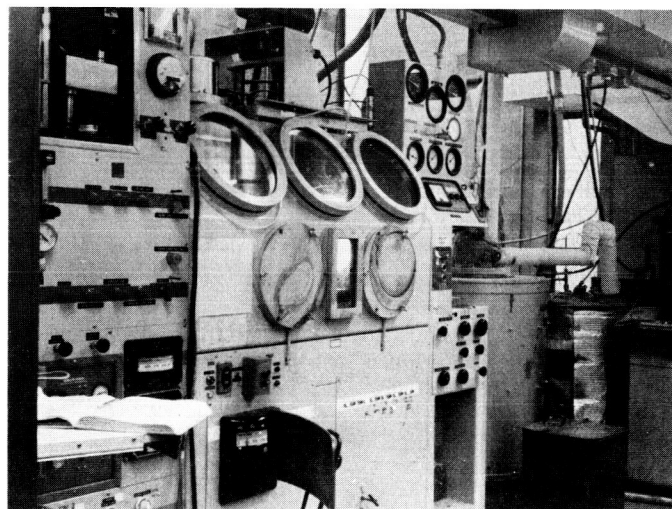
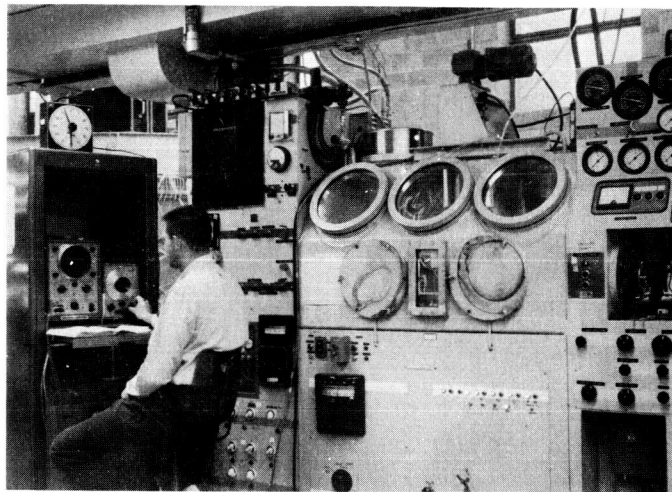


FIGURE 1—HIGH TEMPERATURE ALKALI METALS TEST FACILITY FOR CAVITATION  
DAMAGE RESISTANCE OF REFRACTORY METALS



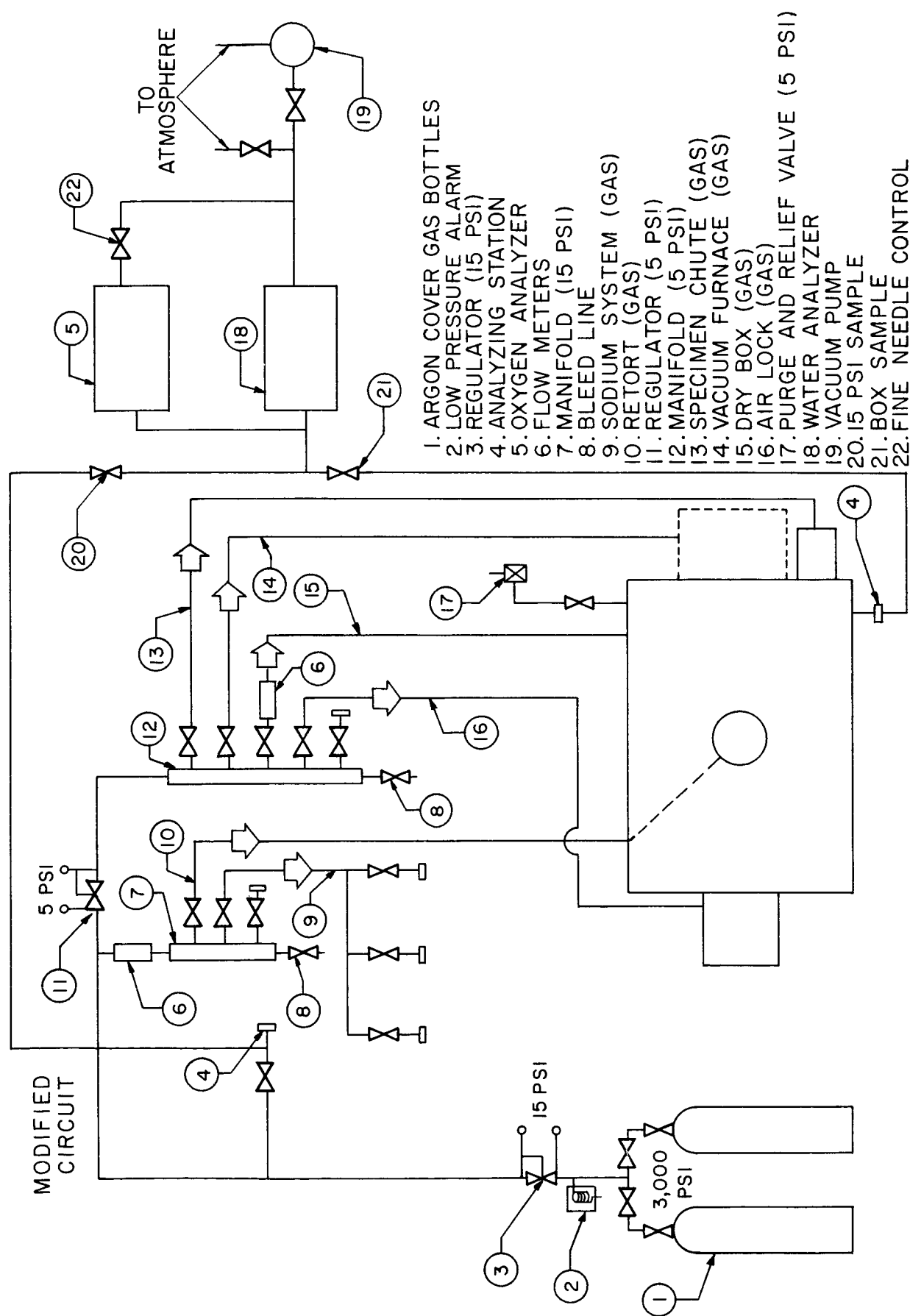


FIGURE 2 - SCHEMATIC DIAGRAM OF MODIFIED COVER GAS SYSTEM

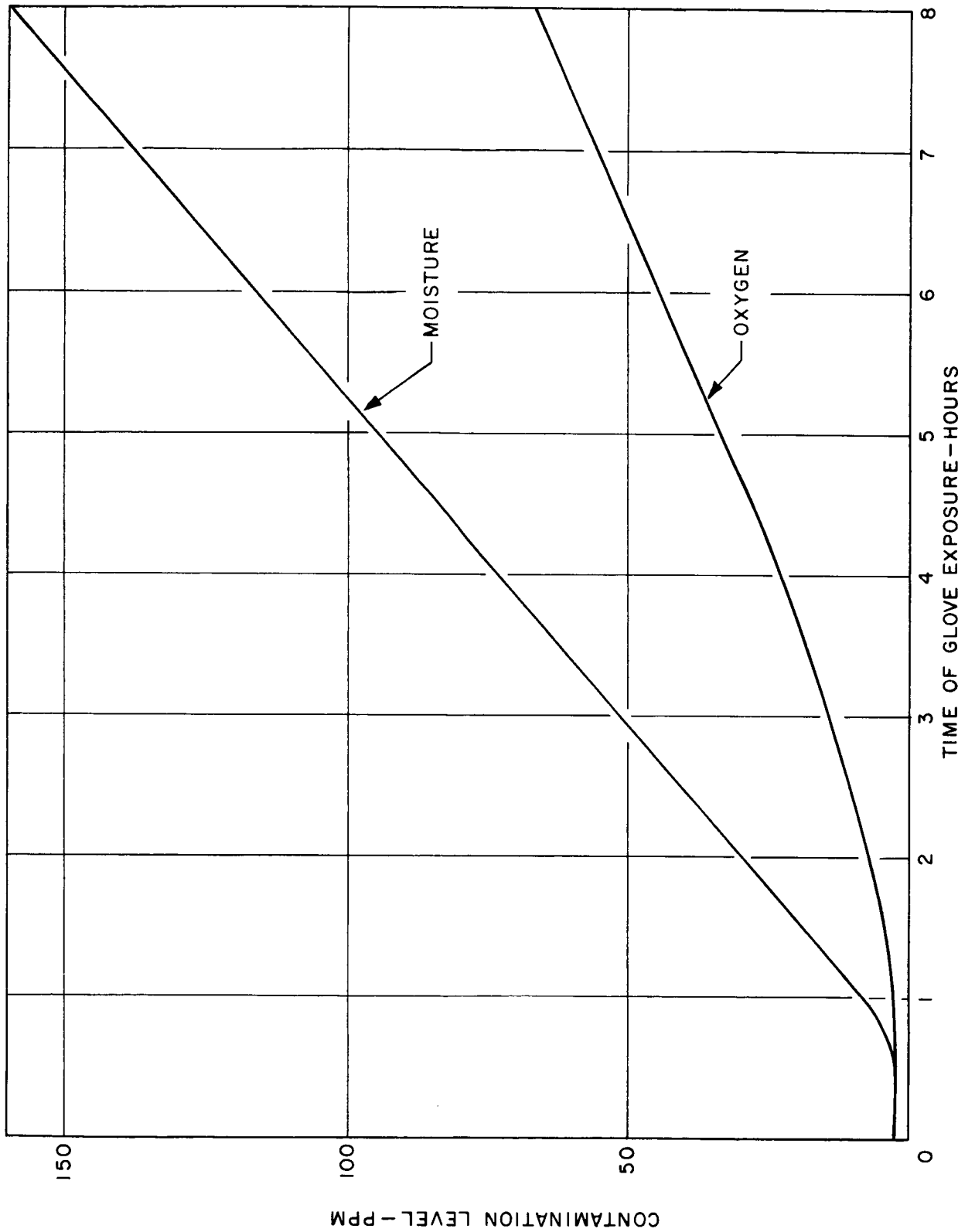


FIGURE 3—OXYGEN AND MOISTURE IMPURITIES IN COVER GAS ATMOSPHERE AS FUNCTIONS OF TIME  
(GLOVE PORT COVERS REMOVED)

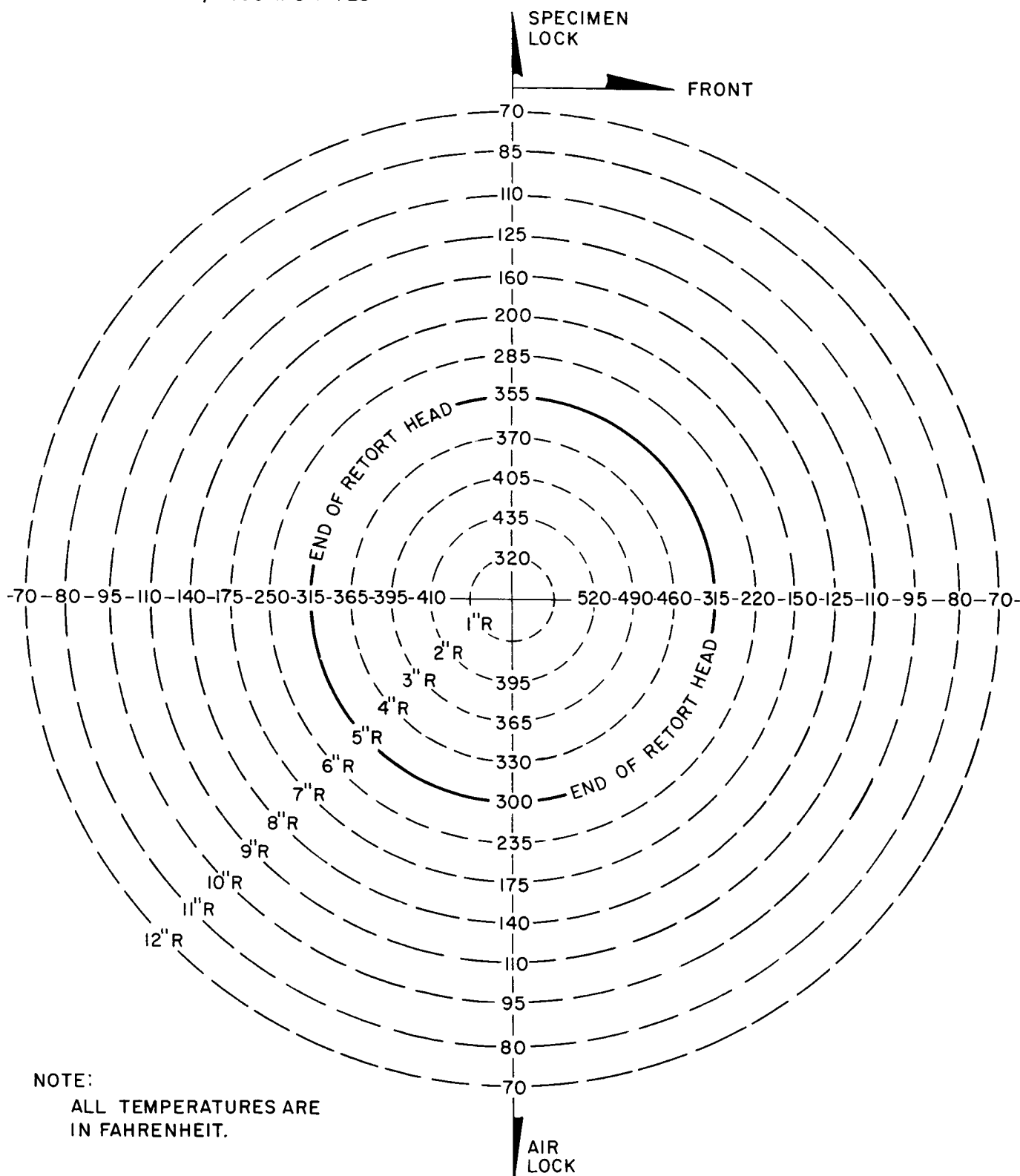


FIGURE 4—VERTICAL VIEW OF DRY BOX FLOOR SHOWING TEMPERATURE GRADIENTS  
AT A RETORT TEMPERATURE OF 1,500° F

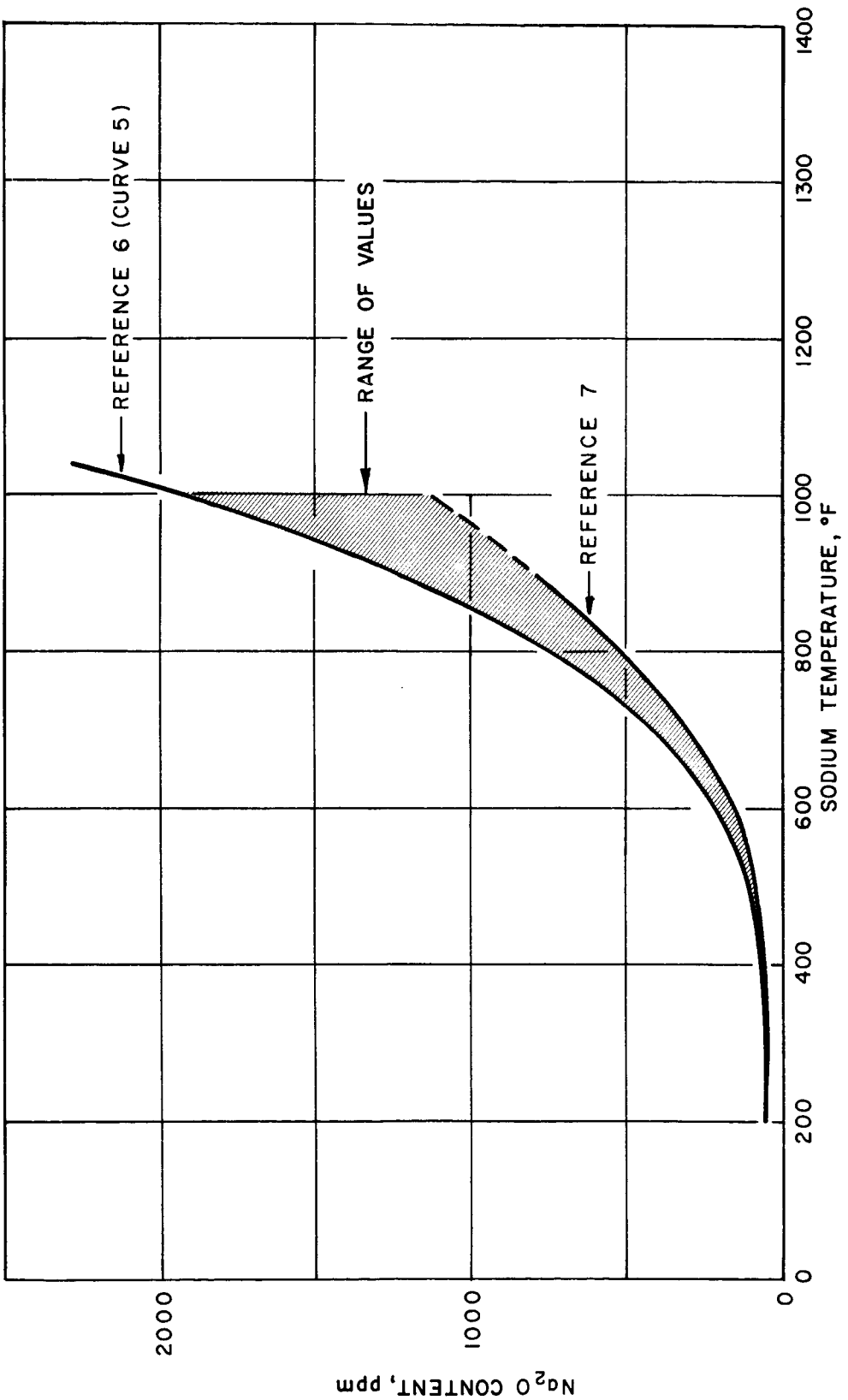


FIGURE 5--SOLUBILITY OF SODIUM OXIDE IN SODIUM

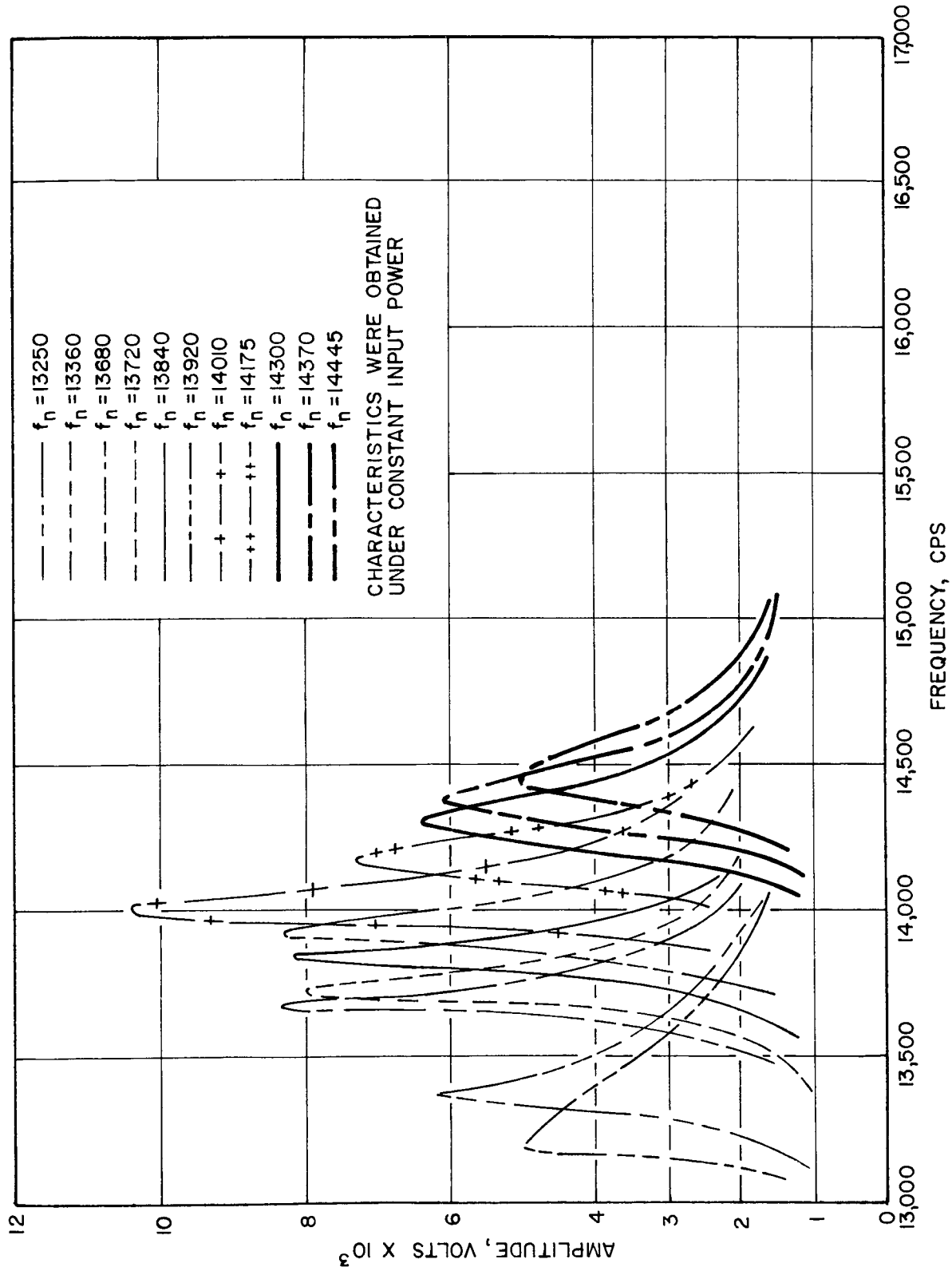


FIGURE 6—CHARACTERISTICS OF THE TRANSDUCER

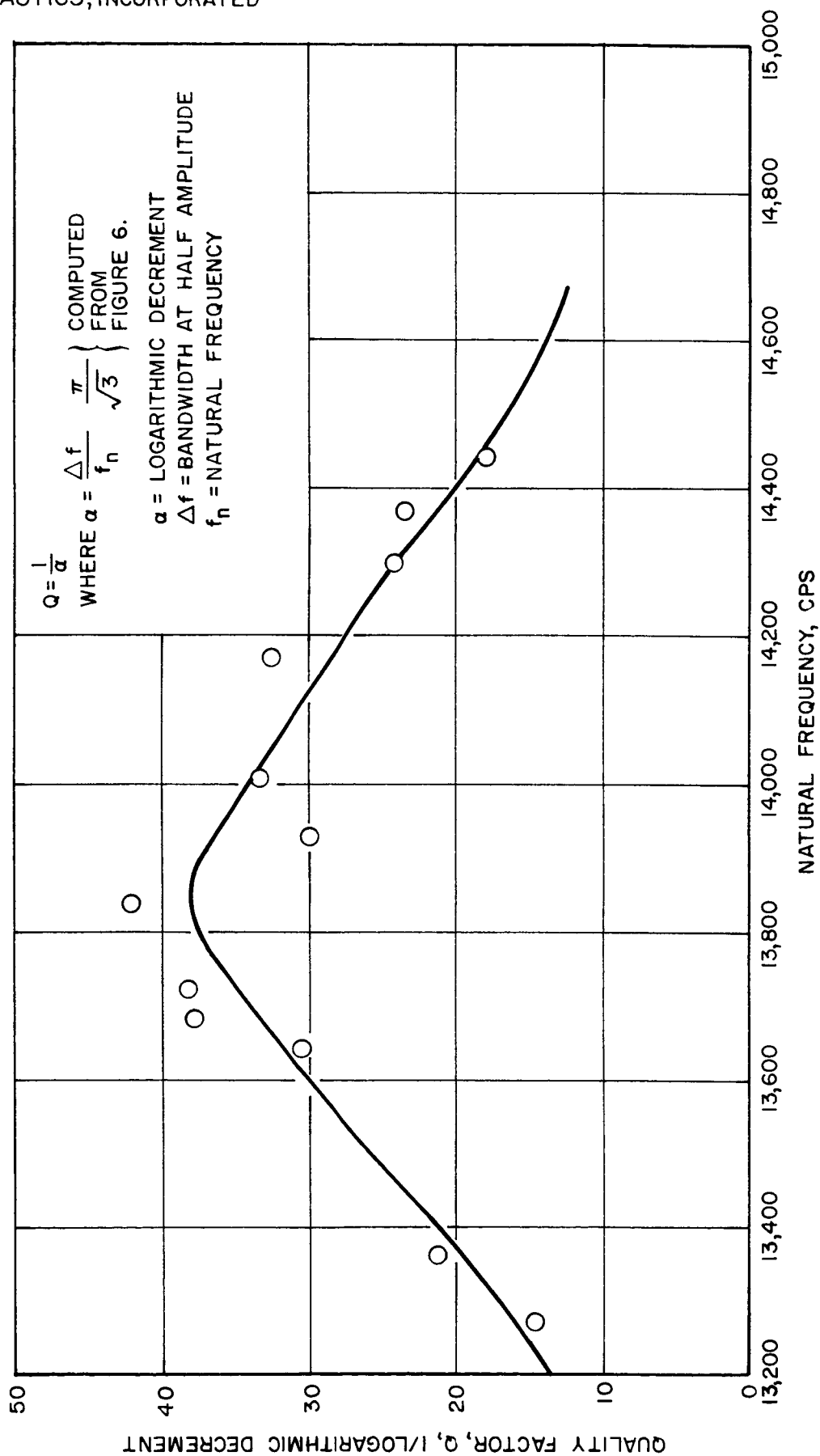
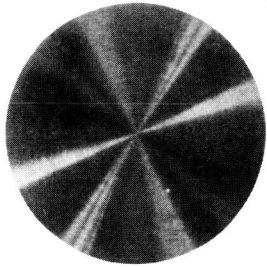
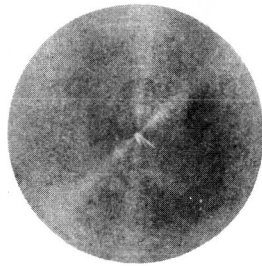


FIGURE 7 -THE EFFECT OF NATURAL FREQUENCY ON THE QUALITY FACTOR

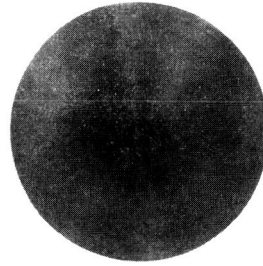
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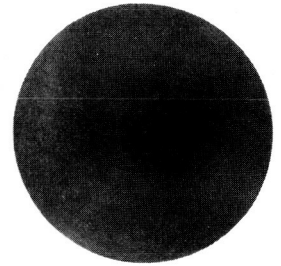
T= 0 MIN.



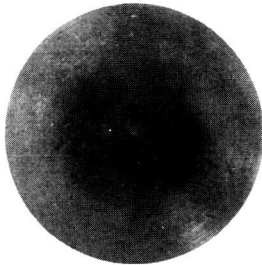
T=117 MIN.



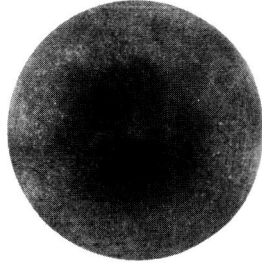
T=177 MIN.



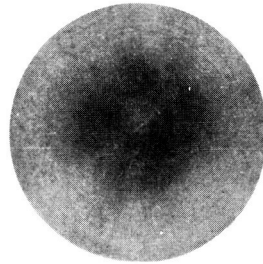
T=237 MIN.



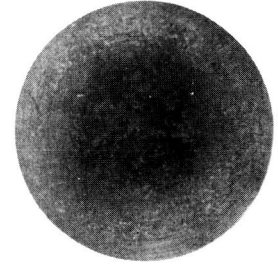
T=297 MIN.



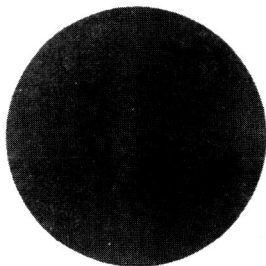
T=357 MIN.



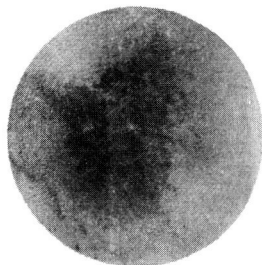
T=417 MIN.



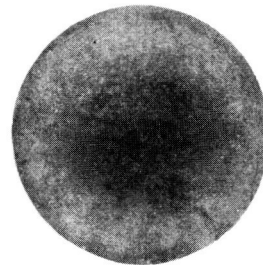
T=477 MIN.



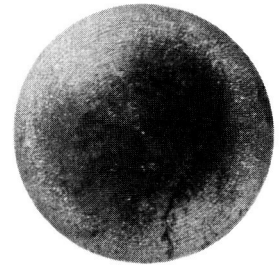
T=537 MIN.



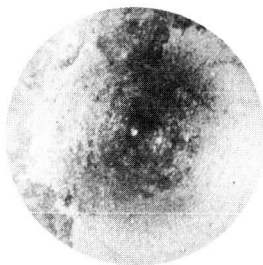
T=597 MIN.



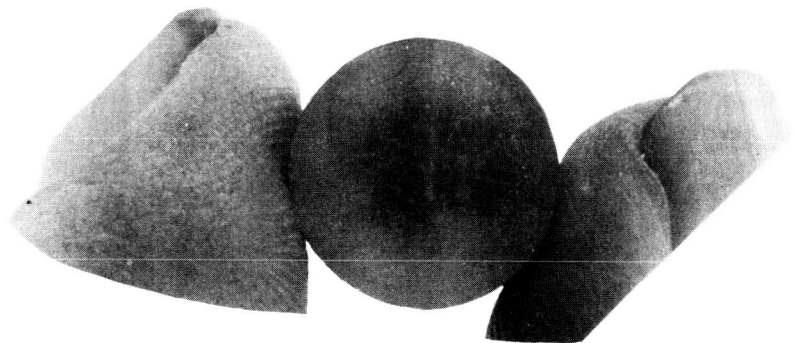
T=657 MIN.



T=717 MIN.



T=777 MIN.



SIZE - 1/2" DIA.

FIGURE 8 - PROGRESSION OF DAMAGE ON THE SURFACE OF 316 STAINLESS STEEL IN SODIUM (1,000°F, VIBRATIONAL AMPLITUDE OF 2 MILS)

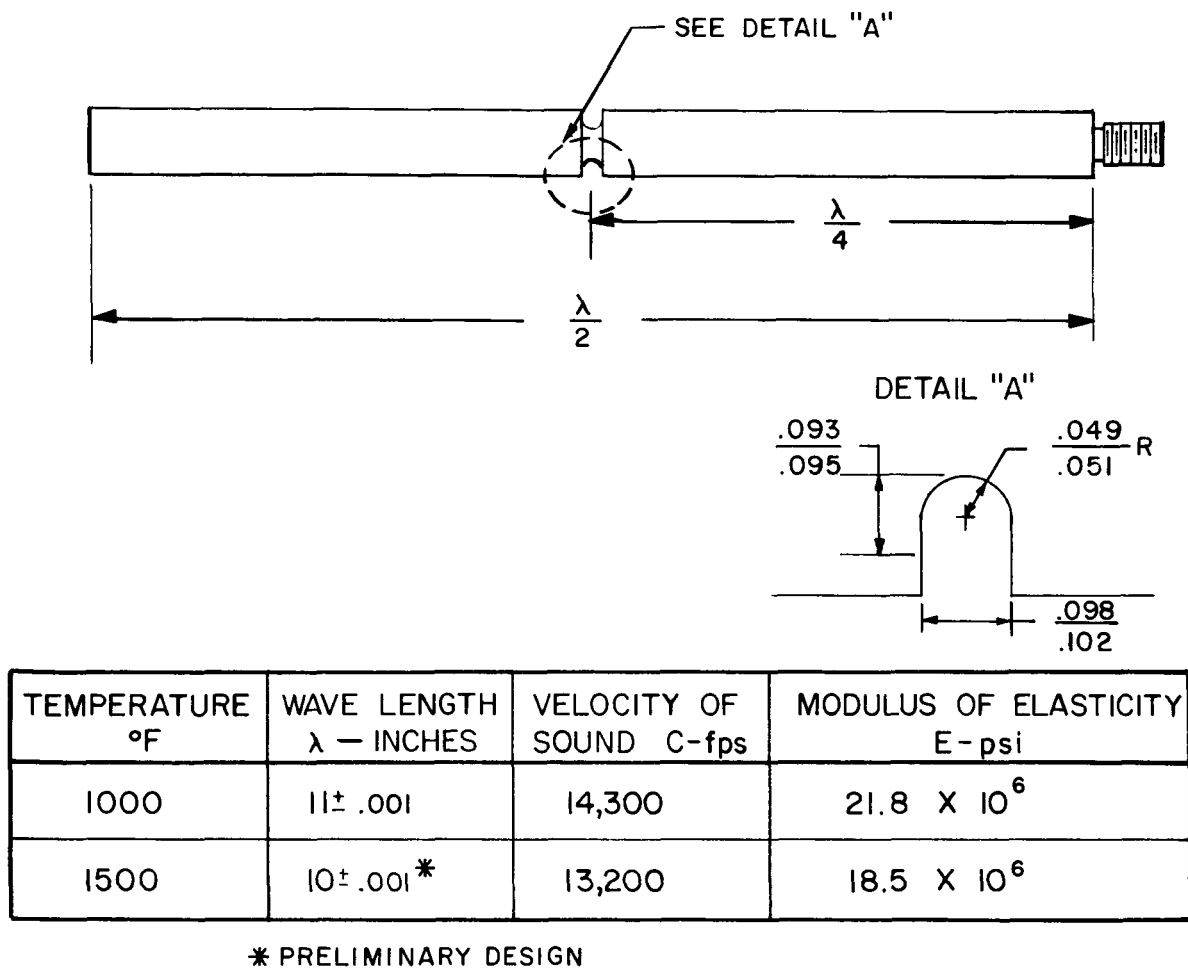


FIGURE 9 - SCHEMATIC DIAGRAM OF HIGH FREQUENCY (14 KCS) FATIGUE SPECIMEN



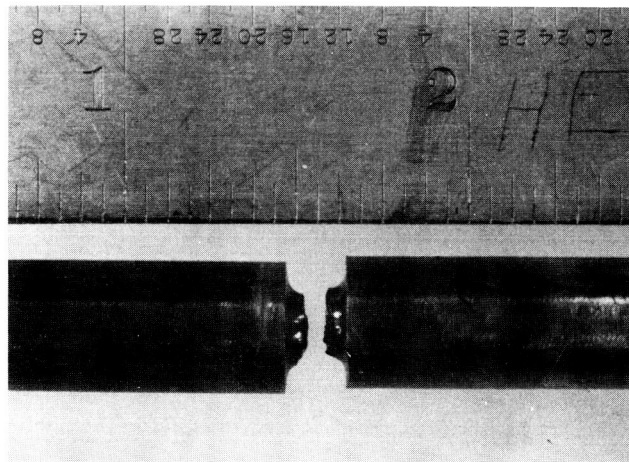
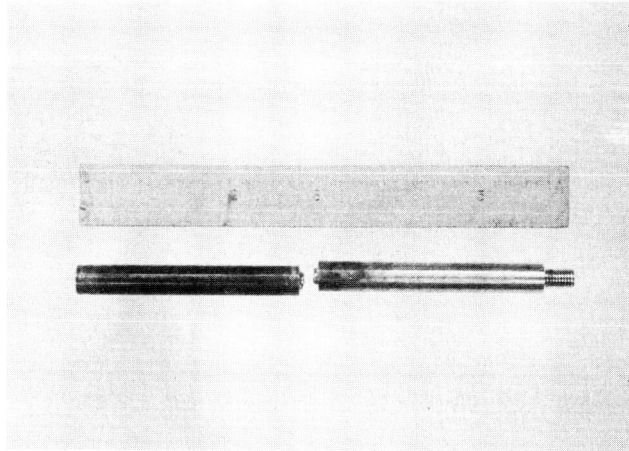


FIGURE 10 - HIGH FREQUENCY FATIGUE SPECIMEN  
(DESIGNED FOR TESTING IN SODIUM AT 1,000°F)

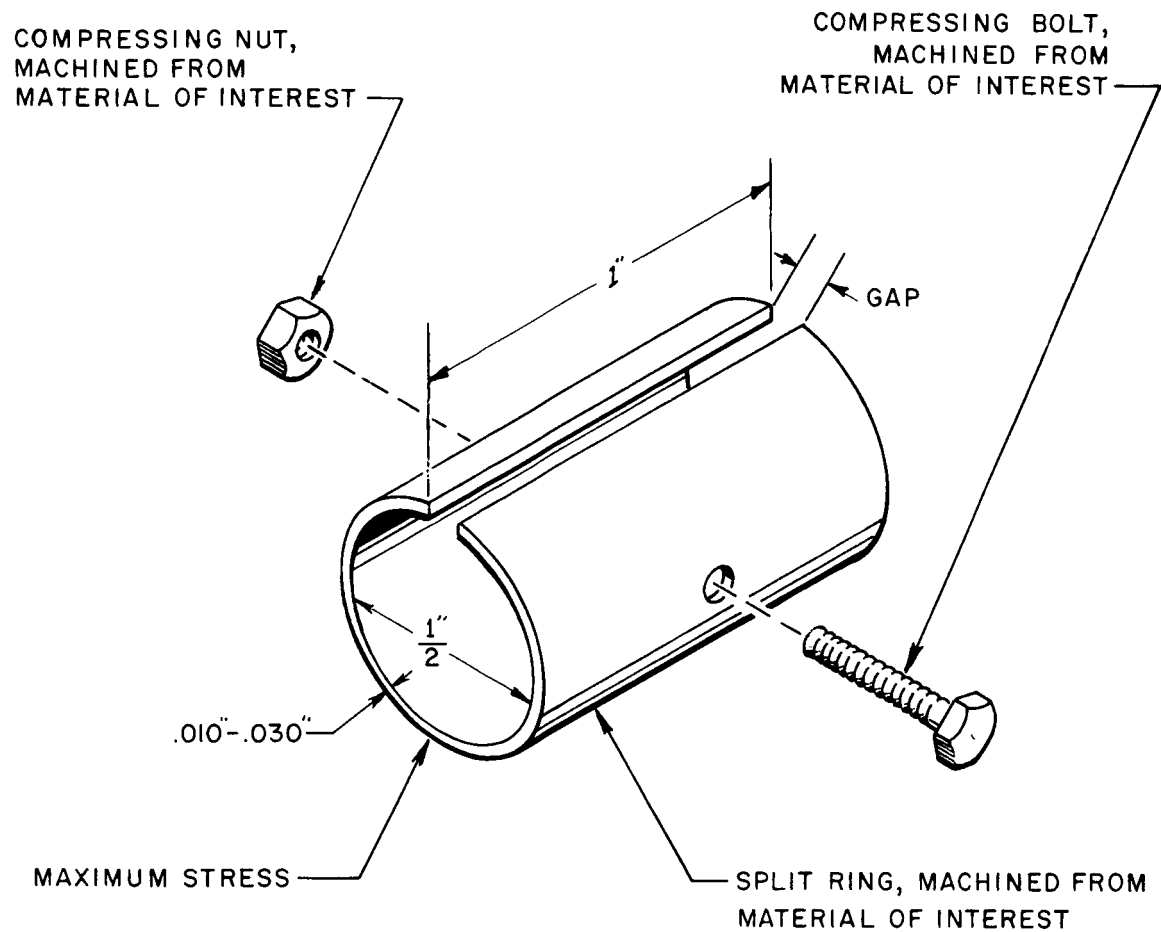


FIGURE II—PROPOSED STRESS CORROSION CRACKING SPECIMEN

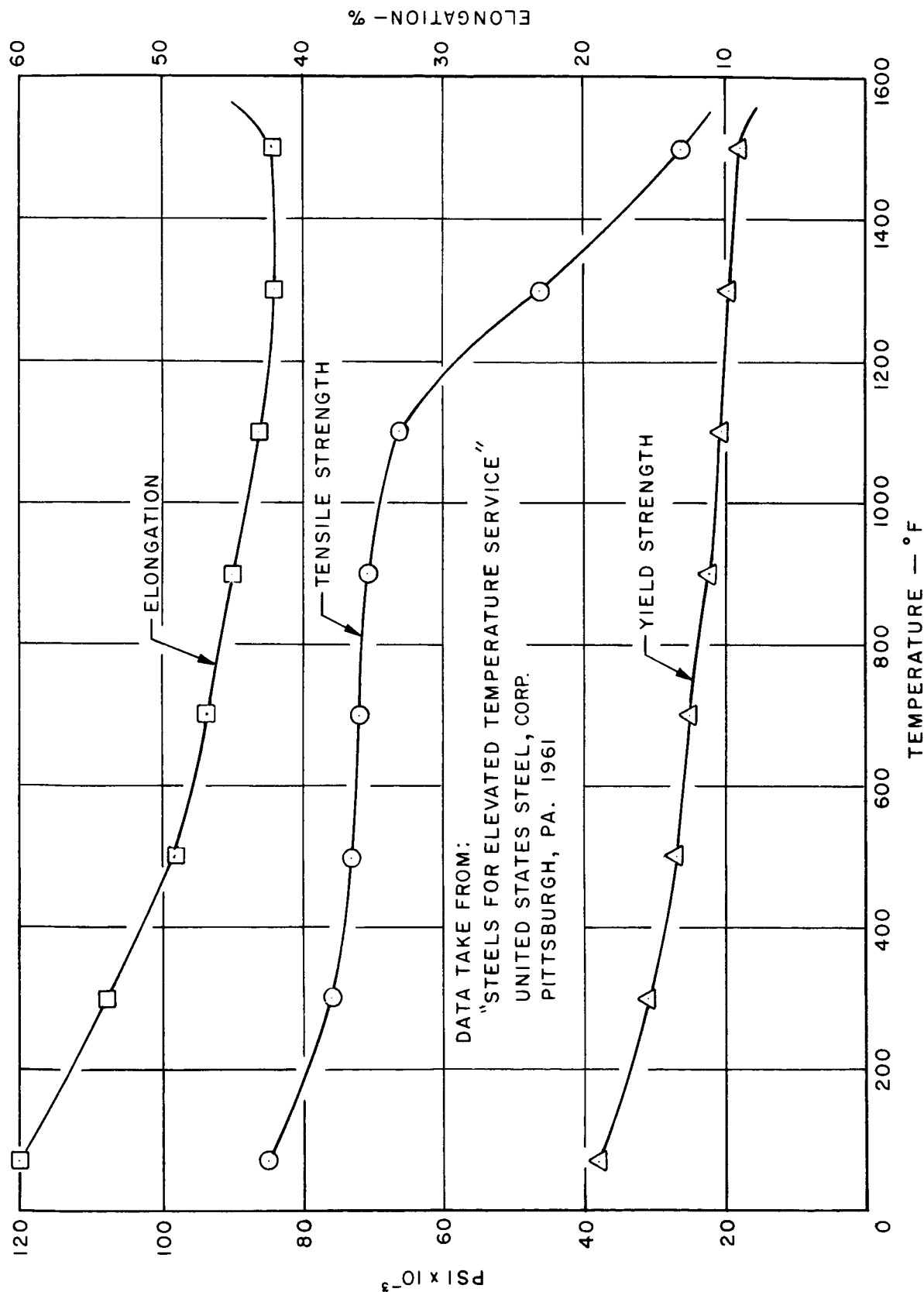


FIGURE 12 — EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES OF 316 STAINLESS STEEL

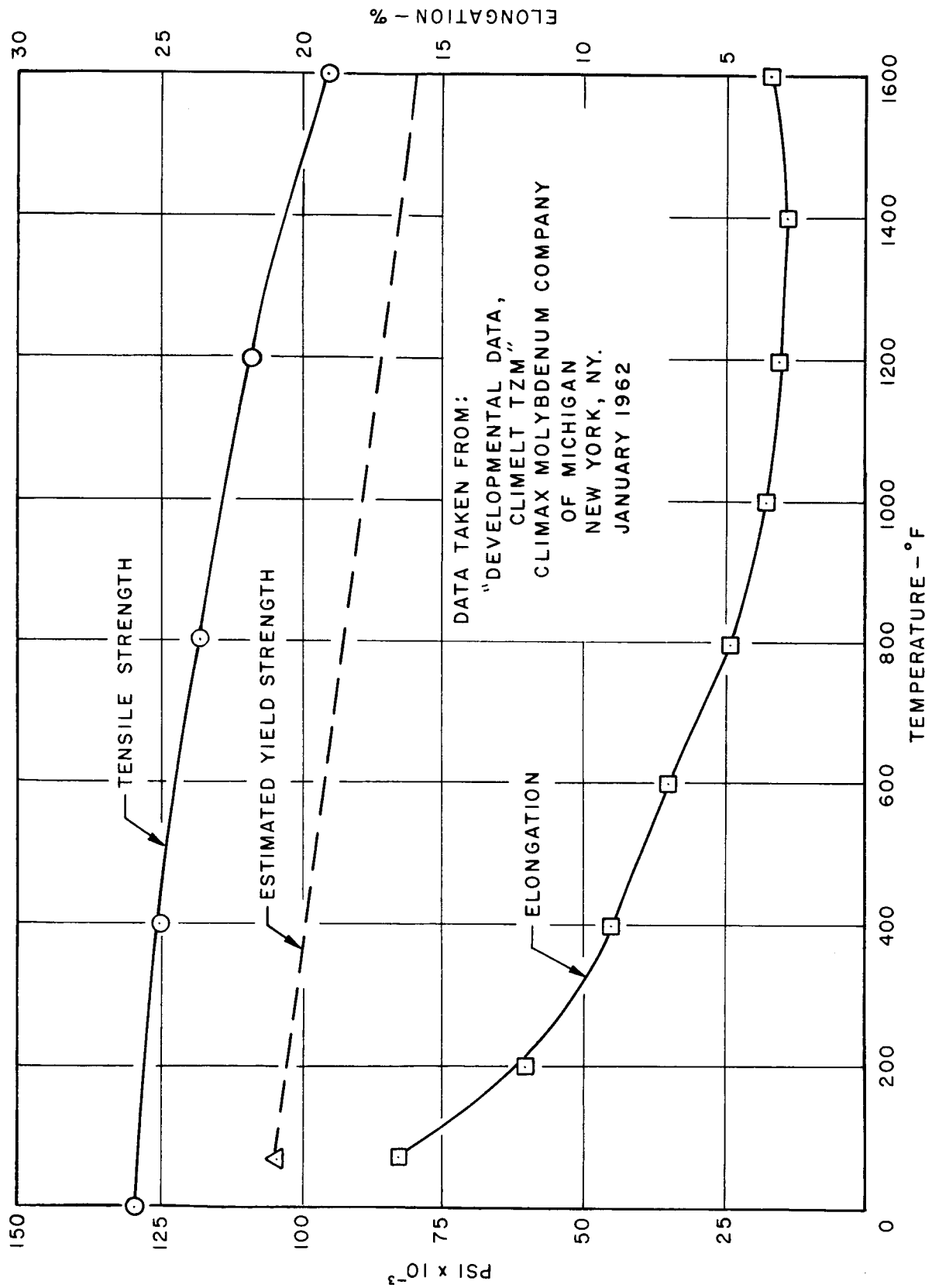


FIGURE I3—EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES OF TZM

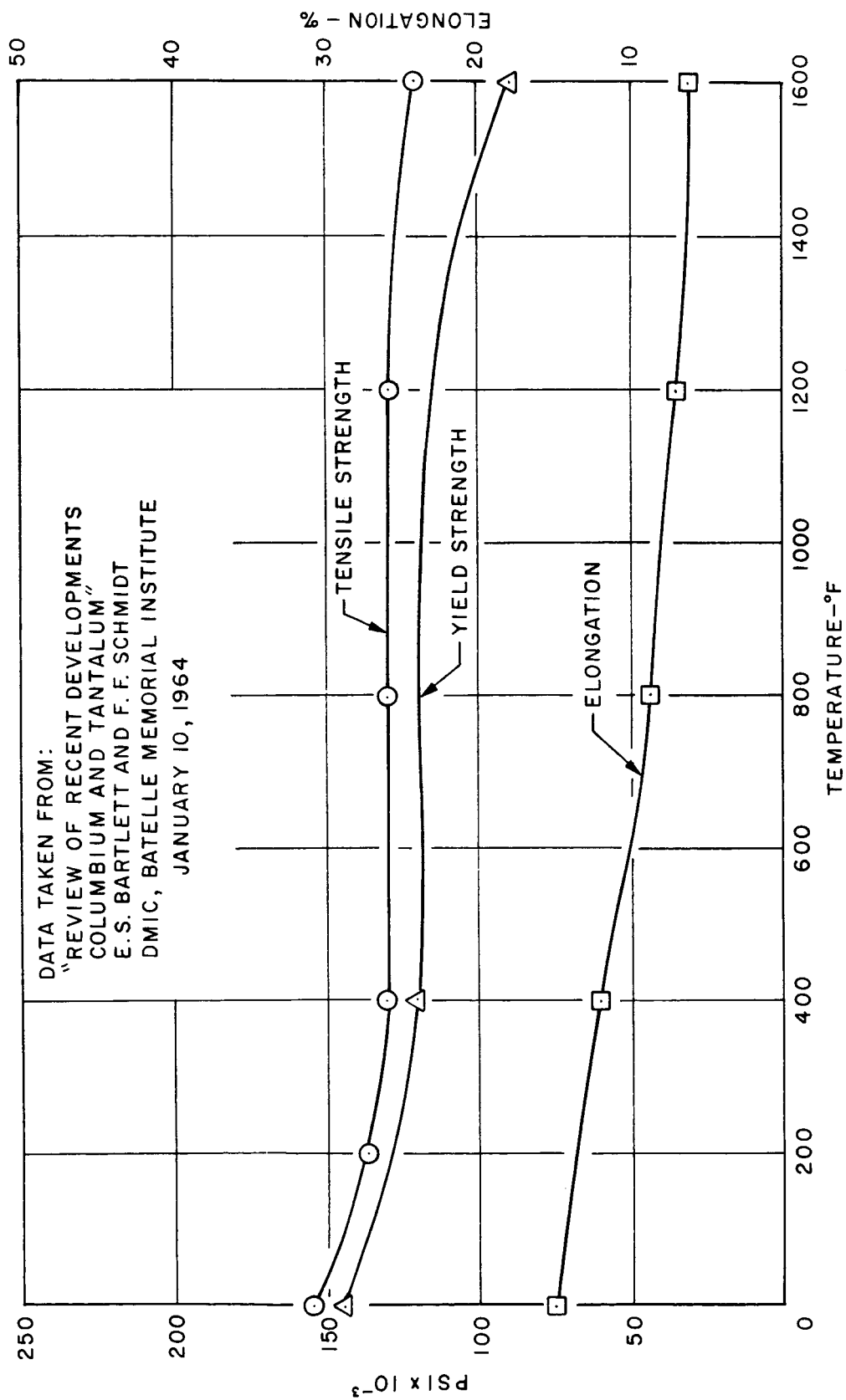


FIGURE 14 — EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES OF T-222

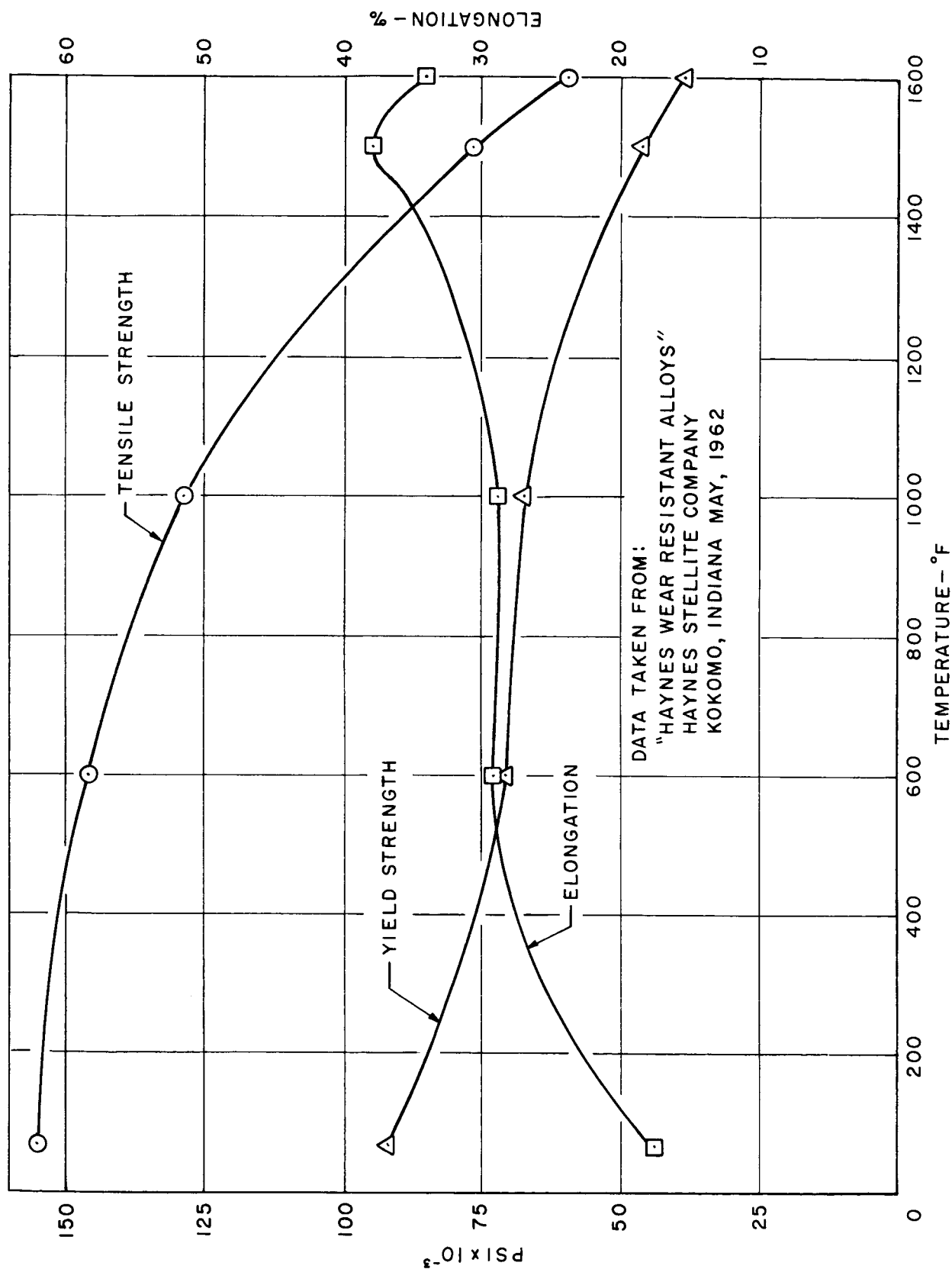


FIGURE 15 - EFFECT OF TEMPERATURE ON MECHANICAL PROPERTIES OF STELLITE 6B

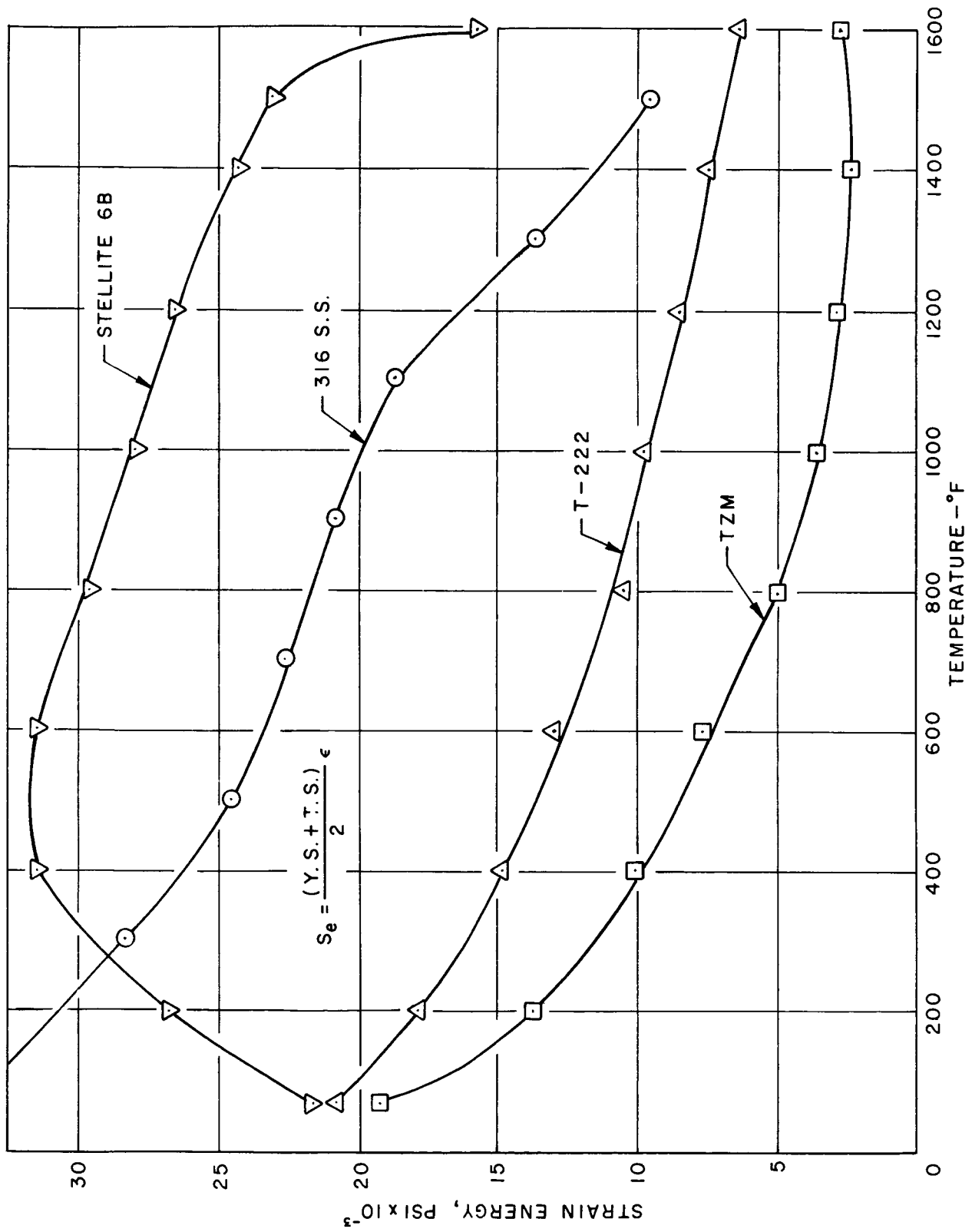


FIGURE 16—EFFECT OF TEMPERATURE ON STRAIN ENERGY